

STRAIN AND DAMAGE MEASUREMENTS IN ION IMPLANTED  
 $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  SUPERLATTICES

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ABSTRACT

Strain measurements in  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  superlattices have been carried out before and after Si ion implantation. For doses up to  $5 \times 10^{15} \text{ cm}^{-2}$ , no atomic intermixing of the sublayers is observed by backscattering spectrometry. However, with x-ray rocking curve measurements, significant changes in the strain profiles are detected for implantations with doses as low as  $7 \times 10^{12} \text{ cm}^{-2}$ . Interpretation of the rocking curves suggests that low-dose implantations release strain in the  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  sublayers. The strain profile recovery of the implanted samples, upon annealing at  $\sim 420^\circ\text{C}$ , implies that the damage caused by implantation is largely reversible.

INTRODUCTION

Superlattices composed of thin alternating layers of different compound semiconductors offer unique possibilities in the conception and fabrication of novel optical and electronic devices [1,2]. To exploit these structures, localized and controlled doping by ion implantation constitutes a desirable processing method. Recently, a few studies have explored ion implantation in  $\text{AlAs}/\text{GaAs}$ ,  $\text{InGaAs}/\text{GaAs}$  and  $\text{GaAsP}/\text{GaP}$  superlattices [3-6]. The primary objective of these studies is to demonstrate that superlattices can be doped by implantation without materially degrading the superlattice. Underlying these studies are the fundamental questions of the nature of the damage created by an ion irradiation in a superlattice, and its evolution upon subsequent thermal annealing.

Almost all superlattices possess strain in one or both sublayers because of the lattice mismatch of the constituents. By measuring this strain as a function of depth, and monitoring its evolution after implantation and annealing, accurate information on the cumulative effects of defects on the superlattice can be obtained. In this paper, the technique of x-ray rocking curves is used to make such measurements. The changes in the rocking curves of  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  superlattices are measured as a function of the dose after Si ion implantations, and also upon thermal annealing. The curves are interpreted in terms of changes in the strain after using the kinematic model of x-ray diffraction [7]. The results help in understanding the onset of damage created by ion implantation.

## EXPERIMENTAL

$\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  ( $x = .88$ ) strained-layer-superlattice (SLS) structures were prepared by metal organic chemical vapor deposition. Alternating layers (10 each) of  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  and GaAs were grown with thicknesses of 140 and 270 Å respectively, on (100) GaAs substrates. Si ions were implanted at room temperature with an energy of 200 keV. To minimize channeling during implantation, the incoming beam was oriented  $\sim 7^\circ$  with respect to the sample's surface normal. The range of the ions is about 2000 Å with a range straggling of  $\sim 900$  Å which places the Si and the damage profiles fully within the superlattice. The implantation doses ranged from  $7 \times 10^{12}$  to  $5 \times 10^{15}$   $\text{Si}/\text{cm}^2$ . Thermal annealing was carried out, on a few samples, in a forming gas atmosphere ( $85\% \text{N}_2 + 15\% \text{H}_2$ ) at  $\sim 420^\circ\text{C}$  for one hour.

X-ray rocking curve measurements were made in the (400) reflection, using a double-crystal diffractometer with  $\text{FeK}\alpha_1$ . The x-ray beam was collimated and rendered nearly monochromatic by a (400) reflection from a high quality (100) GaAs crystal (the first crystal). The beam spot was adjusted to  $\sim 0.5 \text{ mm} \times 1 \text{ mm}$  by slits placed between the first crystal and the sample. The measured rocking curves were fitted with calculated curves using Speriosu's model of kinematical theory of x-ray diffraction in thin epitaxial layers [7]. In this model, the diffraction by the substrate is treated dynamically. For fitting, the structure factors of  $\text{Al}_{.88}\text{Ga}_{.12}\text{As}$  ( $= 117.4$ ) and GaAs ( $= 157.0$ ) were calculated using the tabulated atomic scattering factors [8].

Backscattering spectrometry measurements were also carried out using a 2 MeV He beam. All data refer to room temperature unless stated otherwise.

## RESULTS AND DISCUSSION

Effect of Si Ion Implantation

Figure 1 shows the measured (dashed line) and the calculated (solid line) rocking curves for the as-grown  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  SLS structure. The angle  $\Delta\theta$  is plotted relative to the Bragg angle ( $\theta_B$ ) of the substrate peak,  $P_{\text{sub}}$ , at  $\Delta\theta = 0$ . The reflecting power, plotted on the vertical axis, is normalized with respect to the intensity of the incoming x-ray beam. The periodicity in the structure factors and in the strain of the layers in the SLS generates the subsidiary peaks observed in the rocking curves. The displacement of the peak  $P_0$  from  $P_{\text{sub}}$  measures the average strain in the SLS. For symmetric reflection, as in this case, the equal separation between the subsidiary peaks measures the average thickness of one period in the SLS. The calculated curve in Fig. 1a was obtained using the strain distribution in Fig. 1b. The details of the calculations are given elsewhere [7,9]. From Fig. 1b, the number

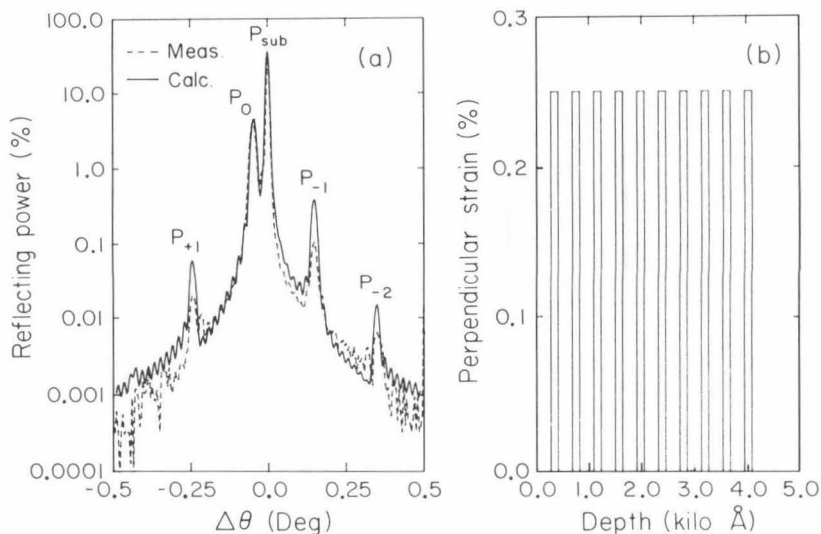


Figure 1 (a). X-ray rocking curves of Fe  $K\alpha_1$  (400) reflection for an as-grown  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  SLS structure with 10 periods. The calculated curve (solid line) was obtained using the depth-strain distribution shown in (b).

of periods in the SLS are verified to be equal to ten. The average thicknesses of the GaAs and the  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  sublayers are 270 and 140 Å respectively. The average perpendicular strains relative to the substrate are 0.0% and 0.25% respectively. The remaining discrepancy between the measured and the calculated curves as observed in Fig. 1a has been attributed to thickness variations in the periods of the SLS and to the nonabrupt interfaces of the sublayers [9]. Thus, from the analyses of the rocking curve measurements, the strain profiles in the SLS are accurately determined.

Rocking curves on the  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  SLS samples were obtained after Si implantations with doses ranging from  $7 \times 10^{12}$  to  $5 \times 10^{15} \text{ cm}^{-2}$  (see Fig. 2). The intensity of subsidiary peaks, except  $P_0$ , diminishes. Also  $P_0$  shifts to an increased angular position  $P'_0$ .

Since the separation between  $P_{\text{sub}}$  and  $P'_0$  measures the average strain in the damaged SLS, the average strain clearly increases after this implantation. Further insight into the change in strain upon implantation is provided by fitting the measured rocking curve with a calculated (solid line) one (see Fig. 2a). To this end, the strains of the sublayers in the model of Fig. 1a were changed and calculations were made iteratively to obtain a best

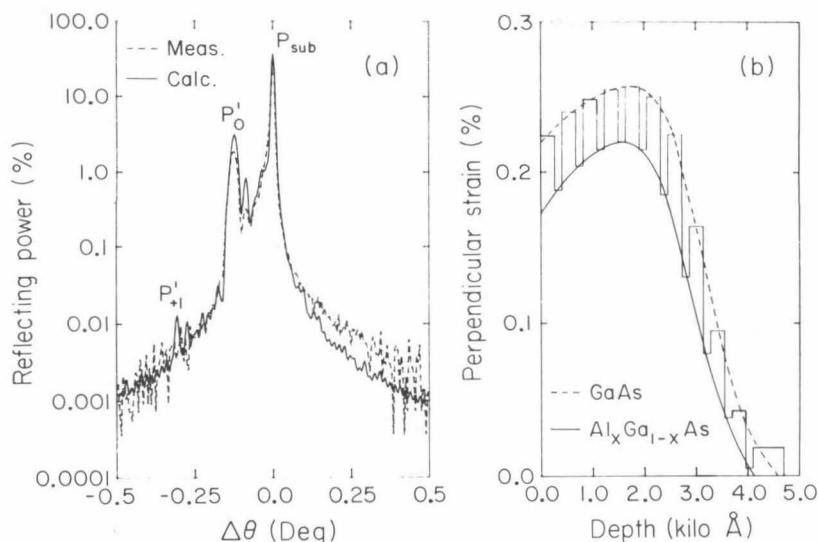


Figure 2. Fe  $K\alpha_1$  (400) rocking curves of an  $Al_xGa_{1-x}As/GaAs$  SLS structure implanted with 200 keV Si ions to a dose of  $7 \times 10^{12} \text{ cm}^{-2}$ . The calculated curve was obtained using the sublayers strain distribution shown in (b).

fit. The strain profile used in this fit of Fig. 2a is shown in Fig. 2b. It is important to note that the damage induced by the low dose implantation considered here reduces the strain in the  $Al_xGa_{1-x}As$  sublayers. On the other hand, the strain increases in the GaAs sublayers, so that the net effect is an overall increase in the average strain of the SLS. A similar increase in the strain of the bulk GaAs after implantation of a corresponding dose has been previously reported [10]; a reduction of strain in a single  $Al_xGa_{1-x}As$  layer has also been measured after implantation with similar doses [11]. The current data are thus consistent with those measurements. It should be pointed out that an equally good fit in Fig. 2a could be obtained by interchanging the strains in the  $Al_xGa_{1-x}As$  and GaAs sublayers in Fig. 2b. This is because of a small difference between the strains in the two sublayers for this particular case. From measurements performed on single  $Al_xGa_{1-x}As$  layers, the strain profile in Fig. 2b appears more probable.

The average strain in the SLS structures increases as the implantation dose rises above  $7 \times 10^{12} \text{ Si/cm}^2$ . The measured average strain as a function of dose is shown in Fig. 3. The average strain initially increases with dose and tends to saturate, to a level of  $\sim 0.44\%$  beyond a dose of  $\sim 5 \times 10^{14}$

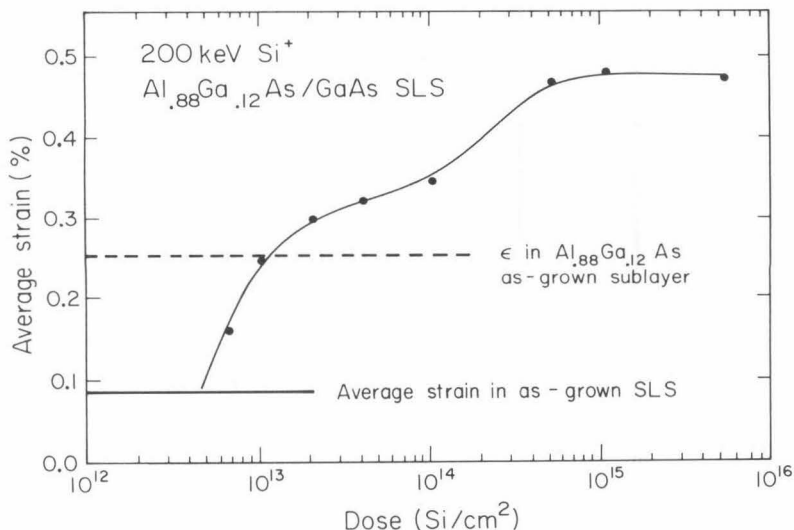


Figure 3. The average strain in the damaged SLS structures as a function of Si ion dose. The average strain was measured by the angular separation of  $P_0^1$  from  $P_{sub}$ , using the relation

$$\Delta\theta = -\langle\epsilon^{\perp}\rangle\tan\theta_B$$

Si/cm<sup>2</sup>. A nonlinear behavior of strain has also been observed in bulk GaAs. Strain measurements carried out on single Al<sub>x</sub>Ga<sub>1-x</sub>As layers on GaAs show that the strain in these layers decreases with doses up to  $1 \times 10^{13}$  Si/cm<sup>2</sup>, and then increases [11]. It would be constructive to pursue such investigations in bulk material to understand the nonlinear behavior of strain with dose in SLS structures.

#### Thermal Annealing

The recovery of the strain profiles of the implanted SLS structures upon annealing is demonstrated in Fig. 4. X-ray rocking curves were obtained before and after annealing at 420°C for one hour of a sample implanted with  $1 \times 10^{14}$  Si/cm<sup>2</sup>. In the curve of the as-implanted sample (Fig. 4a), the angular separation between the peaks  $P_{sub}$  and  $P_0^1$  is about twice that in Fig. 2a because of the higher dose (see Fig. 3). Another striking feature of Fig. 4a is that the peak  $P_{+1}^1$  is more pronounced than in Fig. 2a. The intensity of this peak is related to the difference in the strain between the sublayers of the SLS [12]. The low intensity of  $P_{+1}^1$  in Fig. 2a thus is due to the small difference in the strains of the sublayers, as shown in Fig. 2b.

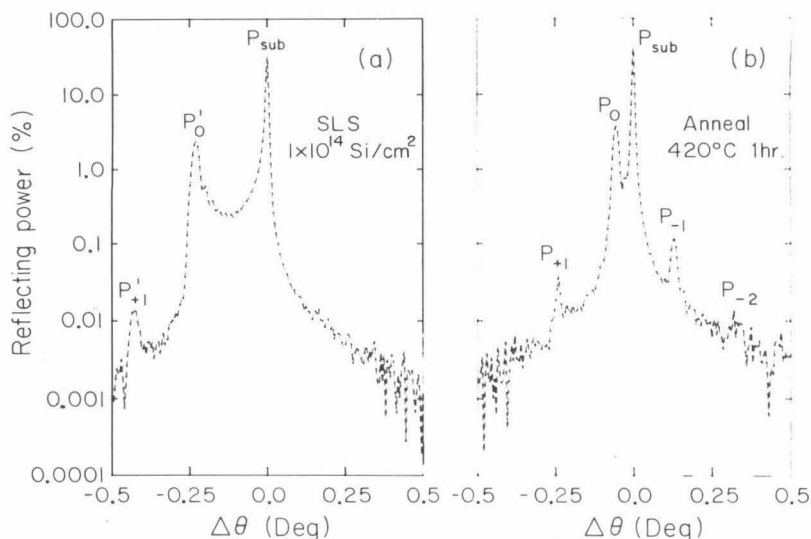


Figure 4.  $\text{Fe K}\alpha_1$  (400) x-ray rocking curves of an  $\text{Al}_x\text{Ga}_{1-x}\text{As/GaAs}$  sample implanted with  $1 \times 10^{14} \text{ Si/cm}^2$ , (a) before annealing (b) after annealing, at  $420^\circ\text{C}$  for one hour.

After annealing, the rocking curve (Fig. 4b) reverts very nearly to that of the as-grown sample (Fig. 1a), which implies an almost complete recovery of the strain profile of the implanted SLS structure.

This investigation establishes that within the resolution of the rocking curves, thermal annealing restores the original state of the SLS. This conclusion was tested by conducting backscattering spectrometry measurements [12]. Insignificant atomic intermixing in depth was observed up to  $5 \times 10^{15} \text{ Si/cm}^2$  after irradiation and subsequent heat treatment. We conclude that the reversible strain alteration produced by the implantation is due to damage generated within the sublayers.

## CONCLUSIONS

Ion implanted  $\text{Al}_x\text{Ga}_{1-x}\text{As/GaAs}$  superlattices have been investigated before and after annealing. Strain profile measurements in these strained-layered-structures have been carried out by x-ray rocking curves. The technique is sensitive enough to detect and measure changes in the strain below 0.1% induced by implantations with doses as low as  $7 \times 10^{12} \text{ Si/cm}^2$ .

The analyses of the rocking curves show that in these SLS structures

the initial stages of implantation reduces the strain in the  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  sublayers. In addition, backscattering analyses establish that up to doses of  $5 \times 10^{15} \text{ Si/cm}^2$ , long-range displacements of atoms are not detectable. This observation is further substantiated by the recovery of the SLS structure upon annealing at a relatively low temperature of  $420^\circ\text{C}$ .

The study of implanted SLS structures, as carried out in this paper, opens up new avenues in investigating the fundamental aspects of ion-solid interactions. In particular, details of damage creation and ion mixing could be clarified by conducting studies similar to this one on highly strained SLS structures with atomically sharp interfaces.

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